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Refractory Metal Alloys and Composites for Space Nuclear Power Systems

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Work performed for

U.S. DEPARTMENT OF ENERGY Nuclear Energy Reactor Systems Development and Technology

Prepared for The Metallurgical Society Fall Meeting cosponsored by The Metallurgical Society of AIME and the American Society for Metals Chicago, Illinois, September 27–29, 1988

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Summary

Space power requirements for future NASA and other United States missions will range from a few kilowatts to megawatts of electricity. Maximum efficiency is a key goal of any power system in order to minimize weight and size so that the space shuttle may be used a minimum number of times to put the power supply into orbit. Nuclear power has been identified as the primary power source to meet these high levels of electrical demand. One method to achieve maximum efficiency is to operate the power supply, energy conversion system, and related components at relatively high temperatures. For systems now in the planning stages, design temperatures range from 1300 K for the immediate future to as high as 1700 K for the advanced sys-NASA Lewis Research Center has undertaken a research program on advanced technology of refractory metal alloys and composites that will provide base line information for space power systems in the 1900's and the 21st century. Special emphasis is focused on the refractory metal alloys of niobium and on the refractory metal composites which utilize tungsten alloy wires for reinforcement. Basic research on the creep and creep-rupture properties of wires, matrices, and composites will be discussed.

Introduction

The objective of our research on refractory metals is to provide an understanding of their behavior and capabilities under conditions that simulate advanced space power system requirements. Current research is focused on monolithic materials to identify alloys that may meet the demands of near term space power components. In addition, refractory metal alloys are being considered for matrices and fibers to be used in metal matrix composites developed for more long term needs that will have to be met in the late 1900's or in the 21st century. These alloys and composites are anticipated to be used in heat generation systems in such applications as cladding for nuclear fuel pins, for heat pipes and tubing, and in energy conversion systems such as the Stirling engine for heater heads, regenerators, pressure vessels, and heat pipes.

The research activities underway on the refractory metals are conducted primarily in-house with some supporting research being done on university grants. The purpose of this paper is to present a summary of our research activities currently underway and to briefly describe the future direction of our research.

Space Power Materials Needs

Current spacecraft require electrical power in the few hundred watts to about 75 kWe range as shown in Fig. 1 (1). The former NASA Skylab operated with a little over 10 kWe of electrical power. In contrast, the space station, NASA's next major space system is anticipated to require nearly 100 kWe initially and to grow to meet ever increasing demands to several hundred kilowatts of electric. Future missions being planned under the Civil Space Technology Initiative (CSTI) and Pathfinder, which include a lunar base or a manned flight to Mars, are expected to push the power requirements to even higher levels as shown in the figure such that tens to hundreds of megawatts of electrical power will be needed.

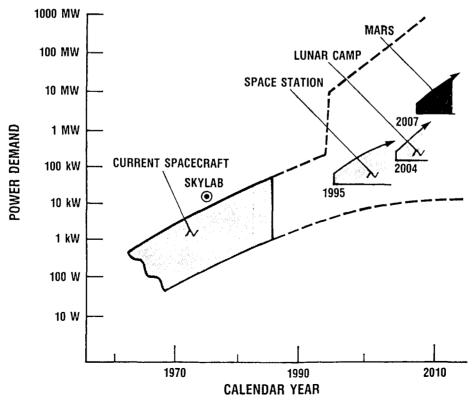
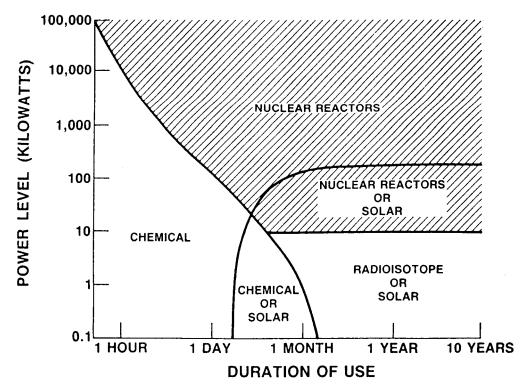


FIGURE 1. - PROJECTED GROWTH IN SPACE POWER.

Possible sources for the power levels described previously are shown in Fig. 2 (2). For lives of over a year and power levels of less than 10 kWe, solar or radioisotope power sources have been used by the United States. In 1965, the United States successfully launched SNAP-10A, the first nuclear reactor to be operated in space. Since then the Soviet Union has used reactors routinely in low, short-term orbits. It can be seen from this figure, that for the 7- to 10-year lives and power requirements anticipated for future systems, nuclear power is the only power source that can be considered.

A joint NASA, DOD, and DOE program to develop a space nuclear reactor capability is currently under way called SP-100 (3), envisioned as a 100 kWe Space Power nuclear reactor; hence, SP-100. The program has focused on demonstrating a Ground Engineering System (GES). This program is managed by the Jet Propulsion Laboratory of NASA and a contract has been awarded to GE to design and build the GES. It is anticipated that the SP-100 type of reactor will be able to support a broad spectrum of space activities that will require large amounts of electrical power including communications, navigation, surveillance, and materials processing. Some of the material related constraints for SP-100 are listed in Fig. 3 and include the use of liquid lithium reactor coolant, 1350 K, 7-year, 1-percent strain design criteria, and a 3000-kg system weight that can be launched by the space shuttle. Based on these constraints, we have undertaken an advanced technology program to allow for future growth of the current power level envisioned for SP-100.

Another high temperature materials need is in the energy conversion system for the high electrical power levels that are being considered. Both the Brayton and Stirling systems are under consideration to convert the heat energy, whether solar or nuclear, into electrical energy (4-5). NASA Lewis has paved the way for Stirling engine technology both for terrestrial and



SP100 SPACE REACTOR SAFETY DOE/NE0083 MAY 1987

FIGURE 2. - POWER SOURCES FOR SPACE APPLICATIONS.



MATERIAL CONSTRAINTS

- LIQUID LITHIUM REACTOR COOLANT
- 1350 K-7 YEAR-1% STRAIN DESIGN CRITERIA
- 3000 kg SYSTEM WEIGHT

FIGURE 3. - SP-100 GOAL: TO DEVELOP NUCLEAR POWER FOR SPACE.

space applications. Shown in Fig. 4 is a cross section of a free-piston space power demonstrator engine that will deliver 25 kWe (6). This engine operates at 1050 K; a lower temperature than the 1350 K that is anticipated for SP-100 and therefore uses a molten salt as the heat source for ground demonstration purposes and is constructed of stainless steels, superalloys, and other materials suitable for low temperature applications. For space applications, refractory metals will be required in such areas as the pressure vessel, heat pipes, heater head, regenerator, and other structural members because of the higher material temperature requirements. The material constraints shown previously in Fig. 3 will have to be met by the refractory metals which will probably be niobium or molybdenum base alloys or composites.

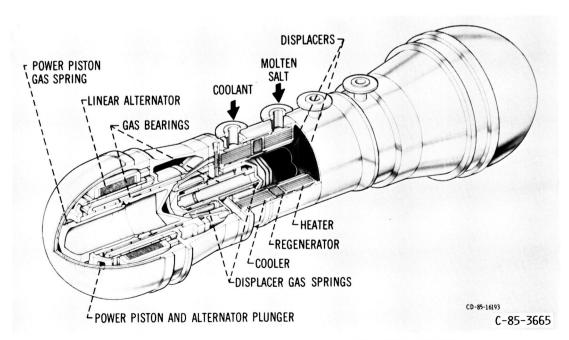


FIGURE 4. - SPACE POWER DEMONSTRATOR ENGINE.

Refractory Metal Alloy Technology

The SP-100 program has selected Nb-1Zr as the nuclear assembly test (NAT) material for the GES. NASA Lewis in concert with Oak Ridge National Laboratory (ORNL) and Westinghouse-Advanced Engineering System Division (W-AESD) is conducting a series of studies to define the creep behavior of this alloy so that design engineers will be able to use this alloy with confidence.

It has been determined from reiterative design considerations that Nb-1Zr has marginal strength for the SP-100 GES. To improve its 7-year 1350 K 1-percent creep strength, such things as a large grain size microstructure (80 um) are being explored. Also heats with increased tantalum and tungsten contents (but still within the allowable specifications) are under test. An alternate approach that we are exploring is to consider a higher strength alloy that will still meet the material constraints for SP-100. We have selected an alloy called PWC-11 with a composition of Nb-1.0Zr-0.1C that was developed in the 1960's. This alloy has been previously tested in lithium and has been shown to be compatible under conditions anticipated for SP-100. However, several question remained to be answered from earlier studies. For example, the weldability of PWC-11 and the subsequent effects on creep properties are not fully characterized, and the long term stability of the carbon precipitates (which are believed to be responsible for the improved strength of this alloy) is not known. We currently have creep tests underway at low stress levels similar to those that may be encountered in SP-100 fuel pin claddings. Both NB-1Zr and PWC-11 are being tested at 1350 K and a stress of 10 MPa. With tests in excess of 20 000 hr, as shown in Fig. 5 PWC-11 has not achieved any practical measurable creep deformation while Nb-1Zr has reached 1-percent creep in 11 000 hr and 2-percent creep in 18 800 hr. The results to date clearly demonstrate the superiority of PWC-11.

The microstructures of Nb-1Zr and PWC-11 are compared in Fig. 6. After the standard 1-hr, 1475-K anneal, Nb-1Zr has an average grain size of 20 μm

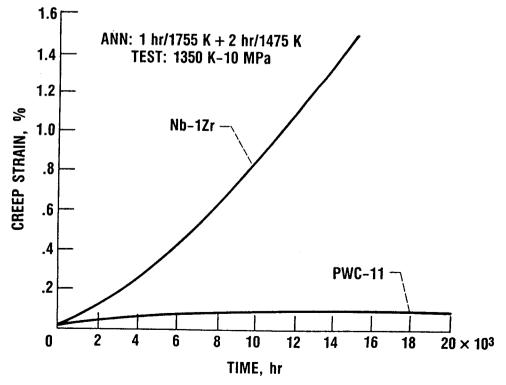
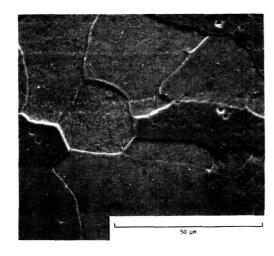


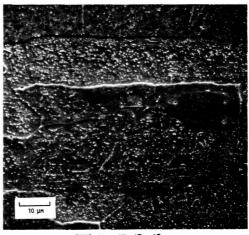
FIGURE 5. - CREEP CURVES FOR Nb-1Zr AND PWC-11.

with a few precipitates present, which are believed to be ZrO2. The microstructure of PWC-11, which had undergone its standard anneal of 1 hr at 1775 K plus 2 hr at 1475 K, is shown in Fig. 6. The annealed material had a mixture of elongated grains with an average grain size of 25 μm measured by the circle-intercept method, with an aspect ratio of approximately 5:1. Numerous shapes and sizes of particles were apparent in the microstructure. The morphology ranges from massive 5 µm particles to submicron needle-like particles which appear to be oriented on slip planes as has been reported previously (7). The majority of particles are believed to be primary carbides which formed during the initial solidification and are neither broken up during the sheet rolling process nor dissolved during the annealing process. A typical microstructure of an EB welded PWC-11 test specimen is shown in Fig. 7. The base metal away from the weld is in the annealed condition. The weldment has a columnar structure with the grain size ranging from about 45 to over 200 μm . The weld zone exhibits extensive second phase precipitation similar to the annealed condition except that the particles appear to be finer and form cell-like domains within the grains.

The high temperature creep strength of PWC-11 (>0.5 T_m), relative to the order of magnitude lower carbon content Nb-1Zr alloy as shown in Fig. 5, has been attributed to the presence of very fine precipitates of (Nb,Zr)₂C and/or (Nb,Zr)C ranging in size from 1 to 10 µm in diameter (8). As with all precipitation-strengthened alloys, the long term beneficial contribution of the precipitate to high creep strength is suspect. It has been postulated that welding and/or isothermal aging of the PWC-11 alloy could result in a significant loss (>50 percent) in elevated temperature creep strength (9). To verify or disprove this postulation, we conducted creep tests in high vacuum (10^{-7} Pa) at 1350 K and 40 MPa to assess the effects of EB welding on creep strength. The creep curves to approximately 1-percent strain are shown for Nb-1Zr and PWC-11 in Fig. 8. The PWC-11 annealed condition (clearly the most creep resistant state) required about 3500-hr to 1-percent strain. A similarly treated sample with an EB weldment required 2125 hr. about a 30-percent decrease in the time for 1-percent strain. A Nb-1Zr specimen was tested in creep for comparison to the annealed PWC-11 alloy. As shown in Fig. 8, the time for 1-percent creep required about 75 hr, a factor of about 45 compared to the annealed PWC-11 and a factor of about 28 for the welded condition. It should be noted that Nb-1Zr was annealed at 1775 K for this comparison which resulted in a grain size of about 45 µm. This larger grain size compared to only 25 µm for PWC-11 should favor a higher creep strength for Nb-1Zr.

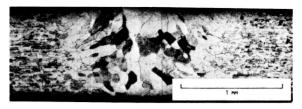


Nb-1Zr ANNEALED 1 HR-1475 K

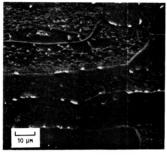


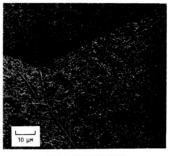
PWC-11 Nb-1Zr-.1C ANNEALED 1 HR-1755 K + 2 HRS-1475 K

FIGURE 6. - MICROSTRUCTURES OF Nb-1Zr AND PWC-11.



ROOT
CROSS-SECTION OF SINGLE-PASS FULL PENETRATION WELD





UNAFFECTED BASE METAL

WELD FUSION ZONE

FIGURE 7. - MICROSTRUCTURES OF ELECTRON BEAM WELDED PWC-11.

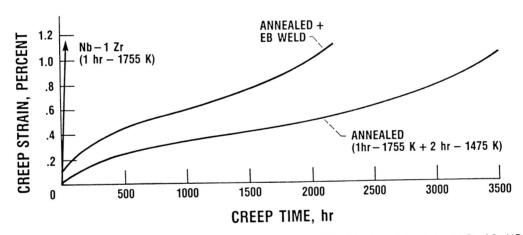


FIGURE 8. - CREEP CURVES OF Nb-1Zr AND PWC-11 AT 1350 K AND 40 MPa.

Moore et al. conducted short-time creep rupture tests to further characterize the effects of EB welding on PWC-11 (10). The EB welds in these tests were perpendicular to the test axis. Tests were conducted in a 10^{-5} Pa vacuum at 1350 K after the post-weld heat treatment (1 hr at 1475 K) and after aging at 1350 K for 1000 hr. In all the creep rupture tests of these specimens, failure occurred in the unaffected base metal (Fig. 9), thus demonstrating that the weld region was stronger.

Based on our creep tests conducted to date, projections have been made for the stress for 1-percent creep in a 7-year time frame and compared to the design requirements for SP-100. The results are shown in Fig. 10. PWC-11 is a factor four times stronger than Nb-1Zr (20 to 5 MPa at 1350 K) over the SP-100 design temperature range of 1350 to 1380 K and affords excellent growth potential over the present SP-100 design stress criterion.



[NECKING ON BOTH SIDES OF WELD]

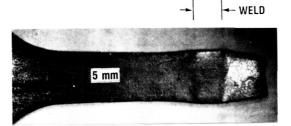


FIGURE 9. - TYPICAL BASE METAL CREEP RUPTURE FAILURE IN EB WELDED PWC-11 MATERIAL TESTED AT 1350 K.

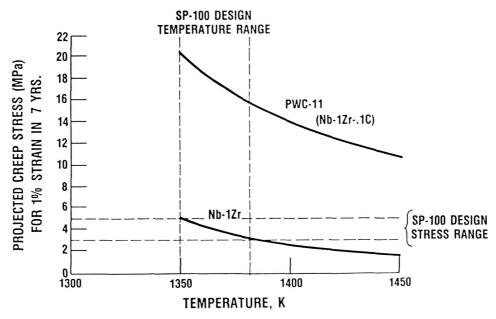


FIGURE 10. - CREEP POTENTIAL OF PWC-11 COMPARED TO Nb-1Zr.

The strength advantage of PWC-11 over Nb-1Zr was explored in more detail by Grobstein and Titran (7). In particular, the concern about overaging of the precipitates during high-temperature exposure for long times was addressed by microstructural characterization of the precipitate (complex carbides) morphology. Several techniques were used including light metallography, scanning and transmission electron microscopy, x-ray diffraction, and chemical analysis of extracted particles. Table I summarizes the results of this study. In the as-rolled condition, the precipitates were relatively coarse, 1 to 10 μm in size, and were found to be hcp Nb₂C. After an initial heat treatment of 1 hr at 1755 K and 2 hr at 1475 K, a different finer precipitate formed. These particles were 0.05 to 0.1 μm in diameter and were determined to be fcc (Zr,Nb)C with the Zr/Nb ratio approximately 70:30. After approximately 5000 hr at 1350 K (0.5 T_m), these fine precipitates almost doubled in size, but did not "overage" and were still effective in pinning dislocations and thus resisting plastic deformation in creep.

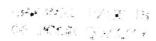


TABLE I. - CHARACTERIZATION OF PRECIPITATES IN PWC-11

(Nb-1Zr-0.1C)a

	As- rolled	After initial heat treatment	After long-term high-temperature exposure
Size, µm	1 to 10	0.05 to 0.1	0.1 to 0.15
Structure	HCP	FCC	FCC
Composition	Nb ₂ C	(Zr,Nb)C	(Zr,Nb)C

^aConclusions: Aging at 1350 or 1400 K with an applied stress does not "overage" the precipitates. After long times (5000 hr) at 1350 K, the precipitates are still effective at pinning dislocations and resisting plastic deformation in creep.

Refractory Metal Composites Technology

The objective of this part of our program is to characterize wires, matrices, and composites for future space power systems where requirements for several hundred kilowatts to megawatts of electricity will need to be met. This advanced technology program focuses on tungsten fibers and Nb or Nb-1Zr matrices and thus can be compared directly with results from the SP-100 GES program on the Nb-1Zr and PWC-11 monolithic alloys. It is anticipated that these composites will enable the technology for advanced space power systems to be more efficient and provide more electrical power by allowing operation at higher temperatures.

Refractory metal alloys have been explored as potential fibers for a variety of matrices (11-12). The creep rupture strength is of primary significance for space power applications since the intended use of the material is for long time operations. A secondary consideration is the density of the fiber since there is a design weight criterion for launching into orbit. The ratio of the 100-hr rupture strength to density for a number of potential refractory metal wires is plotted in Fig. 11 for tests conducted at 1365 and 1480 K. It can be seen that for anticipated growth to higher temperatures, the tungsten-base and molybdenum-base alloys have the superior properties. The strongest alloys, W-Re-Hf-C and Mo-Hf-C are not commercially available so two lower strength tungsten compositions were selected for our initial studies since processing conditions and fiber-matrix interactions can be simulated directly with the tungsten base alloys. The two selected are 218 CS, an example of a lower strength, unalloyed lamp filament alloy and ST300 (W-1.5ThO₂), an example of a stronger, oxide dispersion strengthened alloy. Figure 12 is a plot of the time to rupture as function of stress at 1400 and 1500 K for these two alloys. It should be noted that at the longer times, 1000 to 10 000 hr, the two alloys' strength properties converge at both test temperatures. Based on these results we proceeded to fabricate composites using both compositions in the form of 0.20-mm-diameter wire as the reinforcement fiber material and Nb and Nb-1Zr as the matrix material.

The composites were fabricated using an arc-spray process developed at NASA Lewis which is shown schematically in Fig. 13 (13). In this process, the tungsten alloy fibers were wound on a drum using a lathe to accurately align and space them. The drum was inserted into a chamber which was subsequently evacuated and backfilled with argon. The Nb or Nb-1Zr matrix material in the form of 1.59-mm-diameter wire was arc sprayed onto the drum surface by using a pressurized argon gas stream. After spraying, the coated

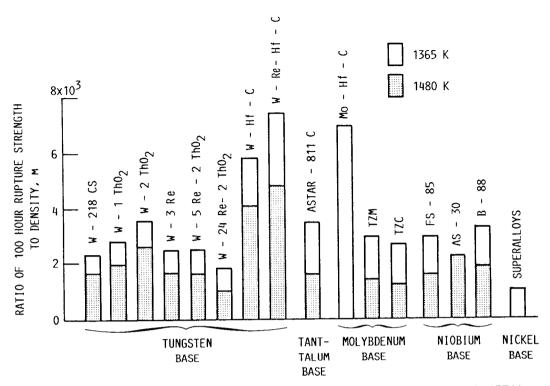


FIGURE 11. - STRENGTH COMPARISON OF CANDIDATE REFRACTORY METAL ALLOY FIBERS.

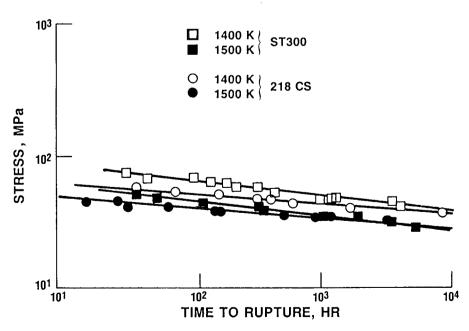


FIGURE 12. - STRESS RUPTURE STRENGTH FOR ST300 (W + 1.5% ThO $_2$) AND 218 CS FIBERS.

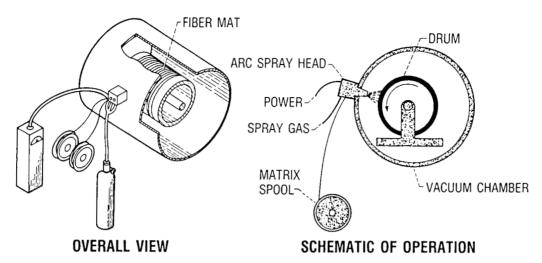


FIGURE 13. - ARC SPRAY MONOTAPE FABRICATION PROCESS.

fiber (monotape) was removed from the drum surface, cleaned, cut to size, stacked in three layers plus matrix only arc-sprayed monotapes on either surface, sealed in a container, and hot isostatically pressed (HIPed) to produce unidirectional fiber-oriented composites. HIP processing parameters were optimized for each combination of fiber and matrix to achieve the best possible properties of the composite. Parameters investigated included temperature, time, and pressure and were varied to explore under what conditions insufficient bonding between fiber and matrix occurred and where excessive reaction took place, as depicted schematically in Fig. 14. A typical microstructure of an as-processed ST300/Nb composite (Fig. 15) indicates minimal reaction between the fiber and matrix during the arc spray and HIP processes. One of the principal concerns in the use of composites for long-term, high-temperature applications is the degree of fiber-matrix interaction. Excessive fiber-matrix reaction could degrade the fiber and thus the composite properties. Figure 16 compares the reaction at the fiber-matrix interface that occurred in ST300/Nb-1Zr composites exposed for

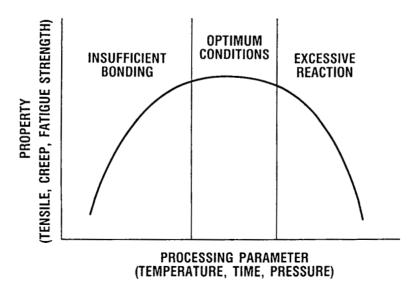


FIGURE 14. - OPTIMIZATION OF FABRICATION PARAMETERS IS CRITICAL TO ACHIEVING MAXIMUM PROPERTIES.

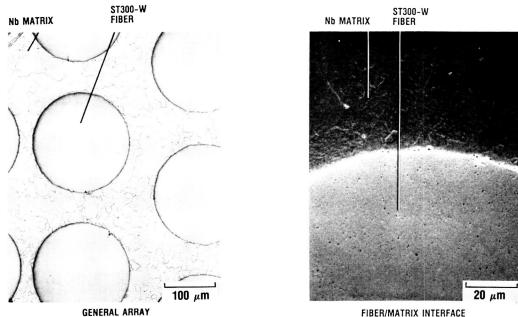
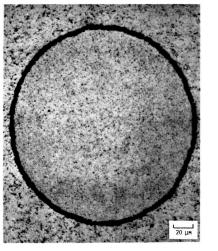
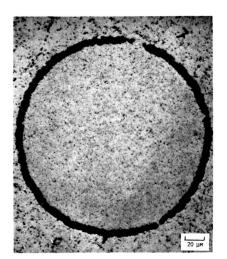


FIGURE 15. - TUNGSTEN FIBER REINFORCED NIOBIUM MATRIX COMPOSITES AS-FABRICATED MICROSTRUCTURE.



EXPOSED 1104 HRS AT 1400 K



EXPOSED 921 HRS AT 1500 K

FIGURE 16. - ST300/Nb + 1Zr FIBER/MATRIX REACTION.

about 1000 hr at 1400 and 1500 K. The depth of penetration into the 0.2-mm-diameter fiber is less than 0.01 mm. The effects of matrix composition on the depth of penetration are compared in Fig. 17 for ST300 fibers in Nb and Nb-1Zr matrices. After 2500-hr exposure the depth of penetration is about 0.01 mm for both matrices. The values for fiber-matrix reactions are in agreement with previously reported diffusion coefficients in the literature for tungsten-niobium diffusion couples (14). Our results thus indicate good microstructural stability for this composite system.

For the creep rupture investigation, tests were conducted on three-ply, unidirectional flat plates from which tensile specimens were cut by electrical discharge machining. An example of a specimen tested to rupture is shown in Fig. 18. Tungsten tabs were TIG welded on both sides of the ends of the composite specimens to prevent specimen shearing at the pin hole locations. It is also possible to make other shapes by the arc-spray, HIP

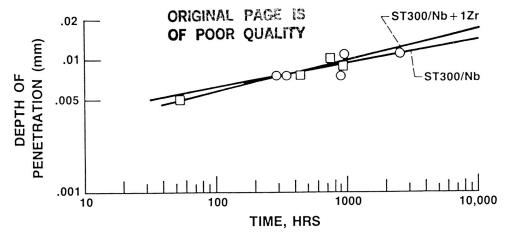


FIGURE 17. - DEPTH OF REACTION PENETRATION VERSUS TIME AT 1500 K.

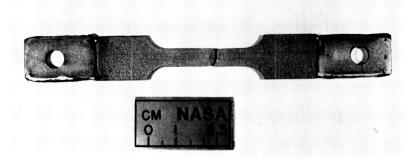


FIGURE 18. - FRACTURED ST300/Nb + 1Zr COMPOSITE CREEP RUPTURE SPECIMEN.

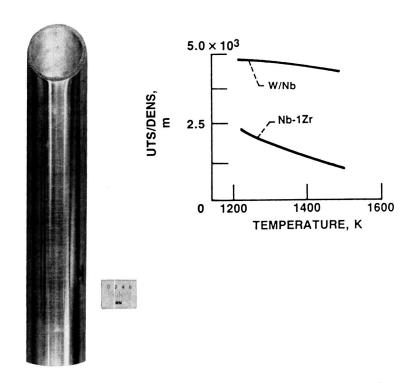


FIGURE 19. - W/Nb COMPOSITE TUBE AND COMPARISON OF TENSILE STRENGTH/DENSITY RATIOS OF W/Nb WITH Nb-1Zr.

process, such as the tube illustrated in Fig. 19. Since for nuclear space power systems, fuel clads, along with heat pipes in tubular geometries will be required, the ability to produce composites having this geometry is very significant. Tensile strengths have been determined for the composites at temperatures of interest to advanced space power systems and are also shown in this figure (15).

The primary property requirement for space power system applications however, is adequate creep resistance for long time exposure. Preliminary creep results on the composite materials have been reported previously (16). A typical creep curve is reproduced in Fig. 20 for a ST300/Nb-12r composite tested at 1400 K under an applied stress of 180 MPa. The creep curves for these composites exhibit the characteristic three-stage creep behavior typical of tungsten and other metals and alloys at elevated temperatures. The strain to rupture ranges from 5 to 7 percent for the composite materials tested in this program.

The fracture surfaces of composite specimens were examined using scanning electron microscopy. Figure 21 shows the fracture surface of a ST300/Nb-1Zr composite where it should be noted that both the fiber and the matrix fail in a ductile manner in creep-rupture testing. Further evidence of fiber- and matrix-ductile behavior is shown in the optical micrograph of Fig. 22, where necking of the fiber and matrix can be observed.

The effective use of fiber reinforcements to increase the creep resistance of Nb and Nb-lZr is shown in Fig. 23. The time to achieve 1-percent creep strain for arc-sprayed niobium under an applied stress of 20 MPa was 17 hr, while arc-sprayed niobium reinforced with 40-vol % ST300 fiber and stressed at an order of magnitude higher stress (200 MPa) has nearly an order of magnitude increase in the time to reach 1-percent strain. Increasing the fiber content results in further increases in creep resistance as shown for the 50-vol % fiber content ST300/Nb-1Zr composite.

Since the reinforcing fibers have a density over twice that of Nb, the $50-vol\ \%$ composite is over one and a half times heavier than niobium, and thus density must be taken into consideration when making property comparisons. A comparison of the creep stress to density ratio for 1-percent

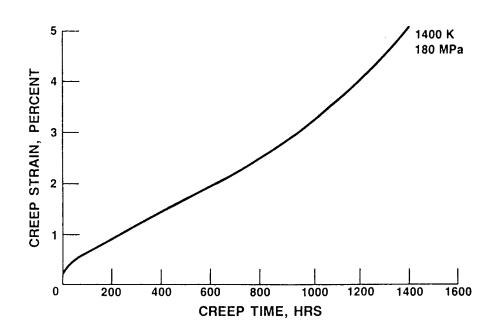
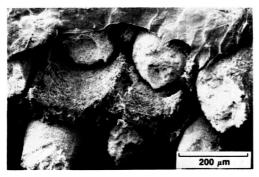


FIGURE 20. - TYPICAL CREEP CURVE FOR ST300/Nb + 1Zr COMPOSITE.

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CREEP RUPTURE TEST DATA:

1500 K 150 MPa 284.5 HRS



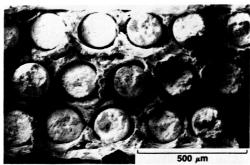


FIGURE 21. - FRACTURE SURFACE OF ST300/Nb COMPOSITE SPECIMEN.

CREEP RUPTURE TEST DATA 1400 K 220 MPa 2325 HRS

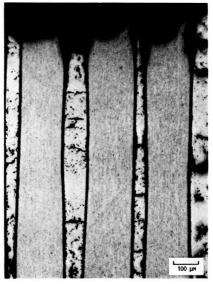


FIGURE 22. - FRACTURE SECTION OF ST300/Nb + 1Zr CREEP RUPTURE SPECIMEN.

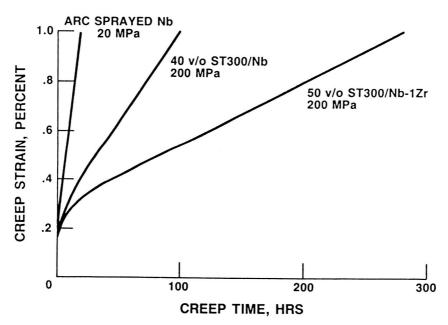


FIGURE 23. - 1400 K CREEP CURVES FOR ST300/Nb AND Nb-1Zr COMPOSITES.

strain for the composites, PWC-11, and Nb-1Zr is made in Fig. 24. On this density corrected basis, the composites are over an order of magnitude stronger than Nb-1Zr and three and a half to four times stronger than PWC-11 at both test temperatures, 1400 and 1500 K.

COMPOSITES NORMALIZED TO 50 VOLUME PERCENT FIBER CONTENT

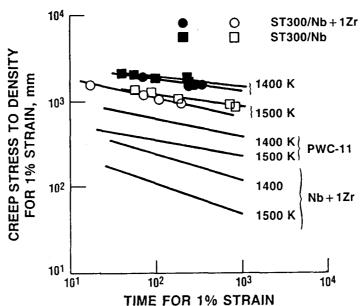


FIGURE 24. - COMPARISON OF CREEP STRESS TO DENSITY RATIO FOR 1 PERCENT STRAIN FOR COMPOSITES, PWC-11 AND Nb + 1Zr.

A comparison of the minimum creep rates of the composites tested at 1400 and 1500 K with that for the arc-sprayed niobium monolithic material tested at 1400 K is made in Fig. 25. It is evident that the composites creep at a much lower rate than the niobium matrix material. Noting that the strain- and strain-rate compatibility must be maintained at the fibermatrix interface during creep of a composite subjected to uniaxial loading, it is possible to estimate the relative magnitude of the stress on the matrix using Fig. 25. For example, it is evident from Fig. 25 that at 1400 K the \$T300/Nb composites exhibit a minimum creep rate of about $1\times10^{-8}~\text{sec}^{-1}$ at 250 MPa. Using the strain-rate compatibility arguments, the data in Fig. 25 suggest that a stress of about 15 MPa would enable the niobium matrix to creep at the same rate. It can be shown using the rule of mixtures, that the corresponding stress on the matrix is only about 3 percent of the total applied stress acting on a composite containing 50-vol % fibers. This means a first order prediction of creep behavior of the composites can be described by the creep equations for the reinforcing fibers. The minimum creep rate of the composites can thus be equated to the power creep behavior as follows:

$$\dot{\varepsilon}_{m} = A exp\left(\frac{-Q}{RT}\right)\sigma^{n}$$

$$\sigma = \sigma_{f} = \frac{\sigma_{C}}{V_{f}}$$

$$\dot{\varepsilon}_{m} = A exp\left(\frac{-Q}{RT}\right)\left(\frac{\sigma_{C}}{V_{f}}\right)^{n}$$

where

 σ_C the stress on the composite

 σ_{f} the stress on the fiber assuming that the fiber carries the total load

- Vf the volume-fraction-fiber content
- Q the apparent activation energy
- n the creep-rate stress exponent
- A a constant for the fiber

The calculated composite creep activation energy Q of 465 to 490 kJ/mol agrees with results for other forms of tungsten tested in this temperature range. It has been proposed that the creep of as-drawn wires occurs by a dislocation mechanism controlled by grain boundary or pipe diffusion (17). The creep-rate exponent n for the ST300-reinforced composites ranged between 5 and 6, which is in agreement with values predicted by simple theories of dislocation climb where n is about 5. It is unlikely that grain boundary diffusion creep or grain boundary sliding controls creep because of the oriented grain structure of the wires.

The relationship between rupture life of the ST300-fiber-reinforced composites and the minimum creep rates is shown in Fig. 26 where a linear inverse relationship was observed of the form:

$$t_R = \frac{C}{\epsilon_m}$$

where C = 0.036. This relationship has been observed in other metallic materials and is known as the Monkman-Grant relationship (18). This relationship was found to be valid for stainless steel composites reinforced with tungsten-thoria fibers and for nickel-coated and uncoated tungsten-thoria wires (19 and 20). The C value of 0.036 observed in this investigation compares favorably with the value of 0.0207 observed for the

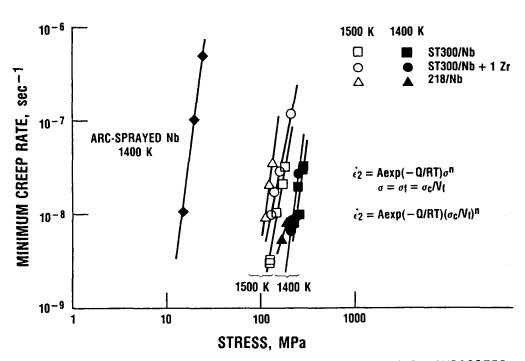


FIGURE 25. - COMPARISON OF MINIMUM CREEP RATE FOR COMPOSITES.

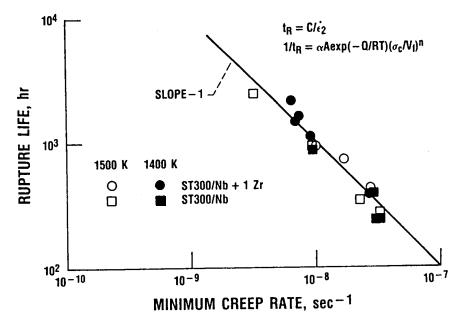


FIGURE 26. - RELATIONSHIP BETWEEN RUPTURE LIFE AND MINIMUM CREEP RATE FOR COMPOSITES.

nickel-coated and uncoated wires. The minimum creep-rate expression (previously described for the composites) can be substituted in the Monkman-Grant relationship to yield an expression which equates the composite rupture life with the stress on the composite and with the volume-fraction-fiber content as follows:

$$\frac{1}{t_R} = \left(\frac{1}{C}\right)A \exp\left(\frac{-Q}{RT}\right)\left(\frac{\sigma_C}{V_f}\right)^n$$

This expression indicates that at a constant applied stress on the composite, increasing the fiber-volume-fraction content will result in increased rupture life values for the composite.

Advanced space power system components will be required to have service and design lives ranging from 7 to over 10 years. In the interest of determining the potential of the composite materials for such applications, extrapolations for both the 1000- and 100 000-hr (11.4 years) density—corrected creep stress to yield 1-percent strain at 1400 and 1500 K and are compared with similar extrapolations for PWC-11 and Nb-1Zr (Fig. 27). These projections show that the composites are an order of magnitude stronger than Nb-1Zr at 1400 and 1500 K. Compared to PWC-11, the composites are five to six times stronger at 1400 K and three to five times stronger at 1500 K. The strength to density values projected for the composites indicate a potential mass savings that could be realized by use of the composites to replace thicker sections of Nb-1Zr. Alternatively, the potential for increased service temperatures and/or service life of components can be considered.

Future Research

The results to date show that for the SP-100 GES, PWC-11 has attractive creep properties that will extend the capabilities of SP-100 compared to a similar system fabricated from Nb-1Zr. However, additional research is needed in the areas of alloy processing, chemistry control, and heat treatment; establishing uniaxial— and biaxial— (tube) creep data bases; long term aging effects in vacuum and lithium; joining process development; and irradiation testing. Our emphasis on advanced materials for future space power

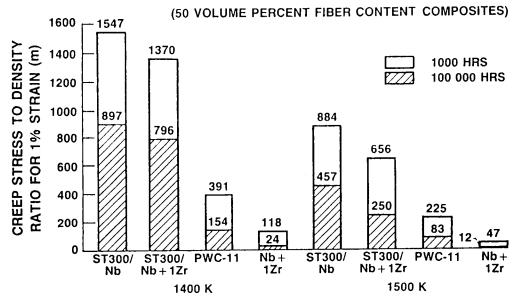


FIGURE 27. - COMPARISON OF PROJECTED 1000 AND 100 000 HR CREEP STRESS TO DENSITY RATIO FOR 1 PERCENT STRAIN.

systems will continue to focus on the tungsten-reinforced niobium-alloys composite materials. Follow-on research will explore the effects of angle plies on creep behavior, fiber-matrix reactions, alternate fibers such as molybdenum base alloys to reduce composite density, and matrix alloying to minimize fiber-matrix reaction.

Concluding Remarks

The results to date from our research in support of the Ground Engineering System for SP-100 and our advanced materials technology program for future space power systems can be highlighted as follows:

- 1. Based on its demonstrated strength advantage, PWC-11 (Nb-1Zr-0.1C) has been selected for the SP-100 reference flight system over the weaker Nb-1Zr alloy.
- 2. Based on creep rupture and compatibility at 1500 K, tungsten fibers are potential reinforcements for Nb-base alloys for space power systems.
- 3. Tungsten-reinforced Nb-1Zr composites provide a ten-fold and a four-fold creep strength advantage over Nb-1Zr and PWC-11, respectively, at 1400 to 1500 K.

References

- 1. J.G. Slaby: NASA TM-100795, National Aeronautics and Space Administration, Washington, DC, 1988.
- 2. SP-100 Space Reactor Safety, DOE/NE-0083, Department of Energy, Washington, DC, 1987.
- 3. R.H. Cooper Jr. and E.E. Hoffman, eds.: Refractory Alloy Technology for Space Nuclear Power Applications, CONF-8308130, Department of Energy, Washington, DC, 1984.

- 4. R.E. English: NASA TM-89863, National Aeronautics and Space Administration, Washington, DC, 1987.
- 5. G.R. Dochat: in <u>Proceedings of the Twenty-Second Automotive Technology Development Contractors' Coordination Meeting</u>, SAE P-155, Society of Automotive Engineers, Warrendale, PA, 1984, pp. 209-213.
- 6. D.R. Hull, D.L. Alger, T.J. Moore, and C.M. Scheuermann: NASA TM-88974, National Aeronautics and Space Administration, Washington, DC, 1987.
- 7. T.L. Grobstein and R.H. Titran: NASA TM-100848, DOE/NASA/16310-6, National Aeronautics and Space Administration, Washington, DC, 1988.
- 8. Advanced Materials Program for November and December 1964, PWAC-1018, Pratt & Whitney Aircraft Corp., Middletown, CT, 1965.
- 9. R.H. Titran, T.J. Moore, and T.L. Grobstein: NASA TM-88842, National Aeronautics and Space Administration, Washington, DC, 1986.
- 10. T.J. Moore, R.H. Titran, and T.L. Grobstein: NASA TM-88892, DOE/NASA 16310-1, National Aeronautics and Space Administration, Washington, DC, 1986.
- 11. D.W. Petrasek and R.A. Signorelli: NASA TN D-5139, National Aeronautics and Space Administration, Washington, DC, 1969.
- 12. D.W. Petrasek: NASA TN D-6881, National Aeronautics and Space Administration, Washington, DC, 1972.
- 13. L.J. Westfall: NASA TM-86917, National Aeronautics and Space Administration, Washington, DC, 1985.
- 14. F.G. Arcella: NASA CR-134490, National Aeronautics and Space Administration, Washington, DC, 1974.
- 15. L.J. Westfall, D.W. Petrasek, D.L. McDanels, and T.L. Grobstein: NASA TM-87248, National Aeronautics and Space Administration, Washington, DC, 1986.
- 16. D.W. Petrasek and R.H. Titran: NASA TM-100804, DOE/NASA/16310-5, National Aeronautics and Space Administration, Washington, DC, 1988.
- 17. S.L. Robinson and O.D. Sherby: <u>Acta Met</u>., 1969, vol. 17, pp. 109-125.
- 18. F.C. Monkman and N.J. Grant: ASTM Proc., 1956, vol. 56, pp. 593-620.
- 19. R. Warren and L.O.K. Larsson: Matrix Composition and Fiber/Matrix Compatibility in W-Wire Reinforced Composites. Proceedings of the Scandinavian Symposium in Materials Science, University of Lulea, Sweden, 1980.
- 20. R. Warren and C.H. Andersson: <u>High Temp. High Press.</u>, 1982, vol. 14, pp. 41-51.

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